THE EFFECT OF HYDROGEN DIFFUSION IN ORTHODONTIC NITI ARCHWIRE

By:

AMIRUL FAHMI BIN AZAMAN

(Matrix no.: 141799)

Supervisor:

Assoc. Prof. Ir. Dr. Abdus Samad Bin Mahmud

July 2022

This dissertation is submitted to
Universiti Sains Malaysia
As partial fulfillment of the requirement to graduate with honors degree in
BACHELOR OF ENGINEERING (MECHANICAL ENGINEERING)

School of Mechanical Engineering

Engineering Campus

Universiti Sains Malaysia

DECLARATION

This work has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any degree.

Signed ……………………………… (AMIRUL FAHMI BIN AZAMAN)

Date: 24/07/2022 …………………………….

Statement 1: This journal is the result of my own investigation, except where otherwise stated. Other sources are acknowledged by giving explicit references. Bibliography/ references are appended.

Signed ……………………………… (AMIRUL FAHMI BIN AZAMAN)

Date: 24/07/2022 …………………………….

Statement 2: I hereby give consent for my journal, if accepted, to be available for photocopying and for interlibrary loan, and for the title and summary to be made available outside organizations.

Signed ……………………………… (AMIRUL FAHMI BIN AZAMAN)

Date: 24/07/2022 …………………………….

ACKNOWLEDGEMENT

First and foremost, I would like to express my sincere gratitude to the Almighty God that gave us this opportunity to pursue this degree in Mechanical Engineering as well as giving me the strength to move forward and completed my Final Year Project. Not to forget, I also would like to express my appreciation to the School of Mechanical Engineering for including the Final Year Project course in our curriculum.

I sincerely express my deep sense of gratitude to Assc. Prof. Ir. Dr. Abdus Samad Bin Mahmud from School of Mechanical Engineering USM for his extraordinary cooperation as well as his invaluable guidance and supervision throughout this report. This thesis is the result of his patience and generous attitude. My Final Year Project could not have managed to meet its completion without the help and support from his knowledge and guidance. The crucial information such as advises on my experimental procedure as well as theoretical knowledge has brought me to this end of my thesis writing.

I owe and respectfully offer my thanks to my senior, Mr Ching Wei who provided me second guidance throughout my journey by giving me endless support and assistance on things that I could not understand. All of the knowledge that has been shared by him, for instance the instruction on using the DSC Analysis Software and also using the UTM Tensile machine. Even when he had the responsibility to take care of his unwell parents, he still provides some time for me and I am truly grateful for all the things that he had done for me throughout my Final Year Project.

I am also thankful to my mother, who continuously give me the support that I need during my four years of study even as a single mother and has helped me to achieve success in every aspect in life. Without her kind devotion, this thesis would have been only a dream for me.

Last but not least, thank you to all the researchers who conducted research related to the studies Nickel-Titanium alloy and hydrogen infusion. I have gained a lot from their findings and has been a huge help for me to conduct my very own experiment. My deepest appreciation to all the people involved in my Final Year Project.

Table of Contents

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

NiTi Nickel-Titanium

SMA Shape Memory Alloy

DSC Differential Scanning Calorimetry

UTM Universal Testing Machine

THE EFFECT OF HYDROGEN DIFFUSION IN ORTHODONTIC NITI ARCHWIRE

ABSTRACT

The application Nickel-Titanium alloy has been widely used in orthodontics because of its superior mechanical qualities, biocompatibility, ductility, corrosion resistance, reduced elastic modulus, and specific features such as superelasticity and form memory effect [1]. When utilized in orthodontic treatments, nickel-titanium alloy is frequently exposed to the oral environment, which commonly causes fractures in the orthodontic wires. In this project, the deformation behavior as well as the thermo-mechanical properties of the NiTi superelastic wires has been investigated immediately after charged with hydrogen and aged at different time intervals. The specimen's mechanical deformation behavior will be tested using the Universal Testing Machine (UTM) while the thermo-mechanical behavior will be investigated using the Differential Scanning Calorimetry (DSC). Experimental results show that compressibility and recovery of orthodontic NiTi wires were dependent on the period which the wires were charged and aged. From DSC analysis, the NiTi wires shows different behaviors with different charging and ageing time. During both the reverse transformation and forward transformation, the height of the peak and number of peaks for samples with different charging time and ageing time are different in comparison with the as-received sample. When charged for 24 hours and aged for 14 days, two peaks were obtained during the martensite to austenite phase transformation which differs from the all the other specimen which indicated two R-phase for that particular phase transformation. Moreover, the deformation behavior obtained from the tests show that specimens that were charged with hydrogen and aged at room temperature produced higher number of deflection for the same value of bending force compared to the as-received specimen. It is speculated that longer hydrogen charging and aging period will allow more hydrogen to diffuse into the wire. This hydrogen diffusion affected the microstructure of the NiTi archwire and caused the reduction in the elasticity of the wire.

ABSTRAK

Aplikasi aloi Nikel-Titanium telah digunakan secara meluas dalam ortodontik kerana kualiti mekanikalnya yang unggul, biokompatibiliti, kemuluran, rintangan kakisan, modulus keanjalan berkurangan, dan ciri khusus seperti keelastikan super dan kesan ingatan bentuk [1]. Apabila digunakan dalam rawatan ortodontik, aloi nikel-titanium kerap terdedah kepada persekitaran mulut, yang biasanya menyebabkan keretakan pada wayar ortodontik. Dalam projek ini, tingkah laku ubah bentuk serta sifat termo-mekanikal wayar superelastik NiTi telah disiasat sejurus selepas dicas dengan hidrogen dan penuaan dijalankan pada selang masa yang berbeza. Tingkah laku ubah bentuk mekanikal spesimen akan diuji menggunakan Mesin Pengujian Sejagat (UTM) manakala tingkah laku termo-mekanikal akan disiasat menggunakan Kalorimetri Pengimbasan Berbeza (DSC). Keputusan eksperimen menunjukkan bahawa kebolehmampatan dan pemulihan wayar NiTi ortodontik adalah bergantung pada tempoh wayar dicas dan penuaan. Daripada analisis DSC, wayar NiTi menunjukkan sifat yang berbeza dengan masa pengecasan dan penuaan yang berbeza. Semasa kedua-dua penjelmaan songsang dan penjelmaan hadapan, ketinggian puncak dan bilangan puncak untuk sampel dengan masa pengecasan dan masa penuaan yang berbeza adalah berbeza berbanding dengan sampel yang diterima. Apabila dicas selama 24 jam dan berumur selama 14 hari, dua puncak diperoleh semasa fasa martensit kepada austenit transformasi yang berbeza daripada semua spesimen lain yang menunjukkan dua fasa R untuk transformasi fasa tertentu. Selain itu, tingkah laku ubah bentuk yang diperoleh daripada ujian menunjukkan bahawa spesimen yang dicas dengan hidrogen dan berumur pada suhu bilik menghasilkan bilangan pesongan yang lebih tinggi untuk nilai daya lentur yang sama berbanding dengan spesimen yang diterima. Diperkirakan selama itu pengecasan hidrogen dan tempoh penuaan akan membolehkan lebih banyak hidrogen meresap ke dalam wayar. Resapan hidrogen ini menjejaskan struktur mikro dawai gerbang NiTi dan menyebabkan pengurangan keanjalan wayar.

CHAPTER 1

INTRODUCTION

1.1 Nickel-Titanium Orthodontic Wire

Nitinol, a nickel–titanium alloy with shape memory and superelastic characteristics, is a smart material. It has a more bone-like elastic modulus than other metal and metal alloy implant materials. Nitinol is used in orthodontics, bone fracture repair, and as bone suture anchors for securing soft tissues like tendons and ligaments to bone. Due to their excellent shape recovery, endurance, and corrosion resistance, nickel-titanium alloys are commonly employed as more practical shape memory or superelastic alloys. When compared to other shape memory alloys (SMAs), NiTi alloys have outstanding workability, yet cold workability is low when compared to traditional metallic materials like steel [2].

The first thing that is needed to be understood are the characteristics of the NiTi archwire. For instance, the shape memory alloy means that the alloy has the ability to sustain large deformation then, they return to their original undeformed shape when stress is removed or with application of heat. The first part of the statement described the pseudoelastic also known as superelasticity which is a reversible elastic response to an applied stress caused by a phase transformation. The other part of the statement for the shape memory alloy explained about the shape memory effect where the alloy is deformed and can return to its original undeformed shape by inducing heat to the alloy.

1.2 Orthodontic NiTi Archwire

Since Nickel-Titanium alloy have some special traits like the shape memory effect and superelasticity, it has been widely used in many sectors including orthodontics. For instance, it has been used as the initial teeth alignment as the alloy provided a medically appropriate moderate continuous force is used to shift the teeth to the right profile after a few months of service life in the oral cavity. However, using the NiTi alloy in orthodontics means that they will be exposed to the oral environment which can affect the mechanical and physical properties. For instance, certain food consumed will interact with human's saliva and produce electricity which will cause hydrogen diffusion in the oral environment. This has been an issue in the orthodontic field since premature fracture might occur to the archwire when there is hydrogen diffusion to the wire. The frictional force between the archwire and bracket slot affects the effectiveness of tooth movement in orthodontic therapy. In orthodontic treatment, the static and kinetic frictional forces produced by various combinations of orthodontic archwires and brackets, as well as the surface topography and hardness of the archwires, all play crucial roles. Sliding mechanics orthodontic therapy includes a relative movement of wire through bracket slots, and frictional resistance arises whenever sliding occurs. Reduced friction force values may be attained when using low-friction brackets, owing to the use of high-performance materials and the ideal shape supplied to the slot and ligating systems [3].

A damaged wire can cause irritation, scrapes, sores, and infection in the mouth, among other things. A broken archwire can cause pain and discomfort by poking into the side of the lips or cheeks. It's also conceivable that a damaged wire will result in wounds and bleeding within the mouth. A cut or sore caused by a broken wire is susceptible to bacterial infection. Symptoms such as fever, increased redness and swelling, as well as growing discomfort or soreness in the afflicted area, may occur as a result.

1.3 Hydrogen Charging and Ageing in NiTi Archwires

NiTi SMAs are frequently used as orthodontic wires to produce a nearly constant force over a long treatment time in order to align and repair a misaligned tooth. However, several NiTi SMA orthodontic wires were discovered to be cracked in the oral cavity after only a few months, indicating that the actual service life is substantially shorter than the planned one. Until date, hydrogen embrittlement has been thought to be the cause of NiTi SMA orthodontic wires' shorter service life. In reality, the protective oxide coating on the surface of NiTi SMA wires is easily destroyed by fluoride in toothpaste and mouthwash during orthodontic therapy [4].

The superelastic and shape-memory characteristics of a SMA NiTi comprising originally a parent-phase structure are known to be altered by the degree of absorbed hydrogen after a few hours of hydrogen charging at ambient temperature. The mechanical characteristics of the NiTi SMA were demonstrated to be altered when the total amount of absorbed hydrogen surpassed 50–200 mass ppm [5]. A considerable drop in tensile strength and the onset of a brittle fracture linked with a stress-induced martensite transition define this deterioration. Nonetheless, the investigations were carried out on the complete specimens using thermal desorption analysis, ignoring the fact that the charged hydrogen was more concentrated at the top layer than in the interior.

The near-equiatomic NiTi alloy retained its superelasticity after a brief time of hydrogen charging, but after 24 hours of ageing in air at ambient temperature, the hydrogen charged NiTi alloy became embrittled. This is due to the fact that the hydrogen in the as-charged specimen is more concentrated at the alloy's sub-surface, and the mechanical characteristics are unaffected. However, during ageing, hydrogen diffuses throughout the specimen and acts as a barrier to the dynamic alteration of the martensite, resulting in fracture [6].

1.4 Problem Statement

Nickel-Titanium alloy used in orthodontic treatments are often exposed to oral environment which usually will inflict fractures on the orthodontic wires. Since orthodontic wires are used in today’s teeth treatment, the wires will constantly be exposed to saliva which contains a certain number of acidities which could inflict small value of electricity when reacted to consumed food hence the production of hydrogen. When nitinol wire is infused with hydrogen, it will disrupt the microstructure of the wire which will cause disturbance in the metallic bond of the wire. To worsen the condition, hydrogen diffuses throughout the specimen during the ageing process and acts as a barrier to the dynamic alteration of the martensite, resulting in fracture.

Nickel-Titanium alloy are used in orthodontic because of its unique traits like the shape memory effect and superelasticity. NiTi archwires can sustain a large deformation before it returned to their initial undeformed shape after stress is removed or by the application of heat. However, there are still some issues nowadays with the use of NiTi alloy in the orthodontic field as it is exposed to the oral environment which could affect the deformation behaviour of the wire. Certain foods, for example, will mix with human saliva and create electricity, causing hydrogen diffusion in the oral environment. This has been a problem in the orthodontic practice because hydrogen migration to the archwire might cause early fracture.

1.5 Objectives of the Project

There are two main objectives in this project:

i. To determine the effect of charging and ageing time toward thermal and deformation behaviour of the Nickel-Titanium orthodontic archwire.
ii. To determine the effect of hydrogen diffusion in bracket bending

1.6 Scope of Project

This experimental study aims to investigate the effect of hydrogen infusion in orthodontic NiTi archwire and quantify the mechanical properties of the wire. The type of wire used for the experiment is Nickel-Titanium rectangular natural super elastic wire (upper 0.016*0.022) and charged with hydrogen before proceeding to the ageing of the wire. In orthodontic field, the dimension and deactivation deflection of the archwires is critically selected by the clinicians to obtain light forces in the occluso-apical. Sample of the orthodontic NiTi archwire will be charged with hydrogen and aged at different periods. Comparison of the thermal and deformation behaviour of the wire can be evaluated using different charging time and ageing time as well as the as-received specimen. By using the DSC analysis, phase transformation temperature of the wire can be obtained while deformation behaviour of the wire can be quantified using the Universal Testing Machine (UTM).

CHAPTER 2

LITERATURE REVIEW

2.1 Characteristics of Nickel-Titanium Alloy

Nickel-Titanium wires or nitinol has been widely used nowadays especially in orthodontics. One of the reasons that allows the NiTi wires to operate efficiently in orthodontics is because of its special traits which is the shape memory effect. The shape memory effect allows the alloy to return to its original shape through heating and this has become a crucial trait in using the nitinol in orthodontics [1]. Nitinols exist in two phases which are the austenitic phase and martensitic phase. The properties of shape memory alloy are greatly depending on the characteristics of the austenite phase which is in body-centered cubic structure as well as martensitic phase in monoclinic structure [7]. Apart from having the shape memory effect as its special traits, the NiTi wires also have superelasticity which means upon unloading the wires, it will return to its original shape before unloading. It is said to be better in deformation where it can deform until 7-8% strain and is forty-times better than the stainless steel capacity [1]. The transformational plateau is a unique property of these alloys, where due stress causes a diffusion less phase changeover from austenite to de-twinned martensite, but SMA substantially enhances elongation at modest stress changes, thus it can provide the optimum biological tooth movement while the crystalline structure of the alloy returns to its original shape as the deactivation starts upon a plateau light forces appreciated [1], [7].

2.2 Nickel-Titanium Alloy in Orthodontic

Nickel Titanium alloy wires have outstanding tensile strength and low load–deflection rates, but their costly price has limited their widespread use. Clinicians were urged to reuse the archwires after recycling due to both cost and the retention of reasonably acceptable elastic characteristics following clinical use. When a wire is repeatedly exposed to mechanical pressures and components of the oral environment for several weeks or months, as well as disinfection between usage, it is considered to be recycled [8]. Whenever the alloy is exposed to the oral environment such as the quantity and acidity of saliva as well as presence of certain enzymes, it will influence corrosion process [9]. This corrosion process does not only happen to nitinol wires but also to the other alloys used in orthodontics such as stainless steel (FeCrNi;SS), cobalt-chromium (CoCr) alloy, copper-nickel-titanium and beta-titanium alloys. The corrosion happens when the metal surface is exposed to a conducting aqueous electrolyte which will encourage the oxidation and reduction (redox) process to happen [9]. Apart from that, diffusion of oxygen in the wires can happen anytime. As the nitinol wire releases nickel and titanium ions, the particles of the oxygen will bond with the titanium ion instead of nickel ion producing the outer coating on the alloy known as Titanium oxide (TiO_2). This is because titanium ions are much more faster compared to nickel ions in ionic bonding. Because of its chemically stable qualities and lack of adverse effects on people, then ceramic TiO_2 has gained a lot of research and has been considered to be the most effective material in organic degradation processes [10]. This is because it can be used as an antibacterial agent based on its photocatalytic properties. An orthodontic patients may be more susceptible to other oral problems. By lowering the odds of oral bacteria adhering to the surfaces of teeth and orthodontic wires, better outcomes can be attained [10]. As a result, photocatalytic TiO_2 surface modification of orthodontic wires may result in more effective orthodontic treatments.

2.3 Effect of Hydrogen in Nickel-Titanium Orthodontic Wire

Many studies have found that immediately after hydrogen charging with a high current density or immersion for a long period, the absorbed hydrogen is concentrated in the archwire's subsurface rather than the core. In the affected zone, this behavior is characterized by an increase in cross-section hardness and a characteristic fragile surface fracture [11]. Diffused hydrogen in the NiTi alloy's austenite phase would enlarge the alloy's volume and impact its transformation temperature. Although it is well known that hydrogen influences many of the features of NiTi, no systematic investigations of how low hydrogen concentrations (650 wppm) affect phase transitions have been conducted too yet [12].

In commercial equipment, the binary equiatomic alloy Ni–Ti is commonly utilized. The pre-martensitic transformation can be used to precede the martensitic transformation in this SMA. The rhombohedral structure of this intermediate phase, dubbed R-phase, is visible [13]. Hydrogen concentrations in NiTi samples ranged from 400 to 1809 wppm were investigated [12] where the phase transitions changed to lower temperatures and the enthalpies of the processes dropped when the hydrogen concentration grew from 3 wppm to 1809 wppm in the baseline material, according to DSC analysis. The 1809 wppm hydrogen content completely suppressed both the austenite and martensite transitions. When using DSC to study the phase transformation behavior of NiTi alloys, one peak appears after cooling and another appears after heating, indicating a one-stage B2-B19# transition during chilling and a reverse transformation while heating [14]. It should be noted that in aged Ni-rich NiTi alloys, the R-phase transition is nonexistent, and multi-stage martensitic transformation is significantly inhibited. R-phase involving B2-R-B19# arises between austenite and martensite as a trigonal phase and another martensite candidate, generating multi-stage transformation behavior.

Hydrogen absorption and consequent embrittlement of Ni-Ti archwires have been documented in the orthodontic literature. It has been hypothesized that hydrogen absorption occurs by interatomic diffusion in a direction from the surface to the bulk material, and that hydrogen accumulates near impure atoms at grain boundaries and dislocations [15]. Furthermore, it was believed that loading increases hydrogen diffusion, most likely due to the larger interatomic space and higher density of dislocations. In-vitro evidence and a case study of a wire failure provided the majority of the evidence. Statistical approach to investigate fatigue in nitinol demonstrated approximately a 2 times increase in 107-cycle fatigue strain limit compared to all of the Standard-grade Nitinol alloys (VAR, VIM, and VIM and VAR)[16] that demonstrated virtually indistinguishable fatigue performance.

2.4 Nickel-Titanium Deformation in Orthodontics

As structural materials are seldom defect-free, and defects may be introduced during service, the damage tolerance of SMAs is critical. Despite the fact that fracture and fatigue crack growth (FCG) characteristics are critical for developing damage-tolerant medical components, there are just a few research on this topic [17]. The very identical fracture toughness of superelastic and martensitic NiTi is explained by the discovery that in the superelastic state, martensite is produced ahead of the crack tip upon loading, independently of whether plane strain or plane stress conditions predominate. As a result, the characteristics of the martensite appear to govern crack formation in the martensitic phase and fracture toughness of superelastic NiTi.

The physical qualities of the material used to fabricate archwire have a significant impact on the force generated by it. Archwires with the same size and form but variable stiffness may now be manufactured due to the vast variety of mechanical characteristics of various alloys. An archwire should ideally be biocompatible, have low stiffness to release mild force upon activation, high strength, and resistance to permanent deformation, maintain its elasticity for a long time, and be simple to use and inexpensive [7][18]. The archwire's load should be able to cause tooth movement in the correct direction with little discomfort. Orthodontic force causes the production of pro-inflammatory cytokines and the formation of inflammatory-like responses, according to research [18]. Prostaglandin’s act as a mediator for tooth movement in orthodontics, but they also improve the transmission of unpleasant stimuli, resulting in pain creation. Orthodontic discomfort can also be caused by the formation of ischemia regions in the periodontal ligament due to sterile necrosis. In orthodontic patients, mild pulp inflammation and allergic responses also lead to discomfort and edema.

Orthodontic study models are necessary for diagnosis, treatment planning, case presentation, and evaluation of treatment outcomes, as well as for the definition of orthodontic malocclusion. These models may be created using a variety of clinic and orthodontist-related procedures. With the advancement of technology, orthodontic models have been converted to digital form, and three-dimensional digital imaging technologies have evolved. With the application and transfer of models to digital media, Yen pioneered the first advancements in the field of digital modelling in 1991 [19]. Traditional means for collecting, storing, and sharing dental information for orthodontic patients' diagnosis and treatment plans are fast becoming digital, and their use is increasing. Some of the common orthodontics problems include overbite, underbite, open bite, crossbite, upper front teeth protrusion and dental midlines not matched [20].

2.5 Effect of Ageing Process in Nickel-Titanium Orthodontic Wire

NiTi alloy retained its superelasticity after charged with hydrogen for a short time, however embrittlement of the hydrogen charged NiTi alloy was discovered after 24 hours of ageing in air at ambient temperature. This is due to the fact that the hydrogen in the as-charged specimen is concentrated on the alloy's sub-surface, and the mechanical characteristics are unaffected. However, following ageing, hydrogen diffuses throughout the specimen and acts as a barrier to the dynamic transition of martensite, resulting in fracture. It is worth noting that, despite the minor improvement in hardness, embrittlement happens in the austenite–martensite transition stage when the entire specimen is impacted by hydrogen diffusion. To test this theory, a microhardness test was performed on the cross-sections of the loaded and unloaded specimens until around 2.5 percent deformation was achieved at a low strain rate of 105 s^{-1}, immediately after 16 hours of charge (Point A, Figure 1b). In comparison to the as-charged alloy after 16 hours, the microhardness profile reveals that the hardness is not restricted to the subsurface of the alloy. Indeed, after 16 hours of immersion, even in the core of the specimen, the hardness values are somewhat greater than the immediately examined specimen, where the hardness is equivalent to the non-charged one (Figure 2). When compared to the immediately tensile-tested specimen after charge, the material exhibits embrittlement over a shorter length of hydrogen charging time after 24 hours of ageing at room temperature. Furthermore, this embrittlement is readily visible when the hydrogen distribution is almost uniform throughout the specimen, indicating that hydrogen embrittlement is dependent on the hydrogen distribution. From the researched that was previously done, it was indicated that after ageing, the embrittlement of the NiTi took place as the whole wire was affected [6].

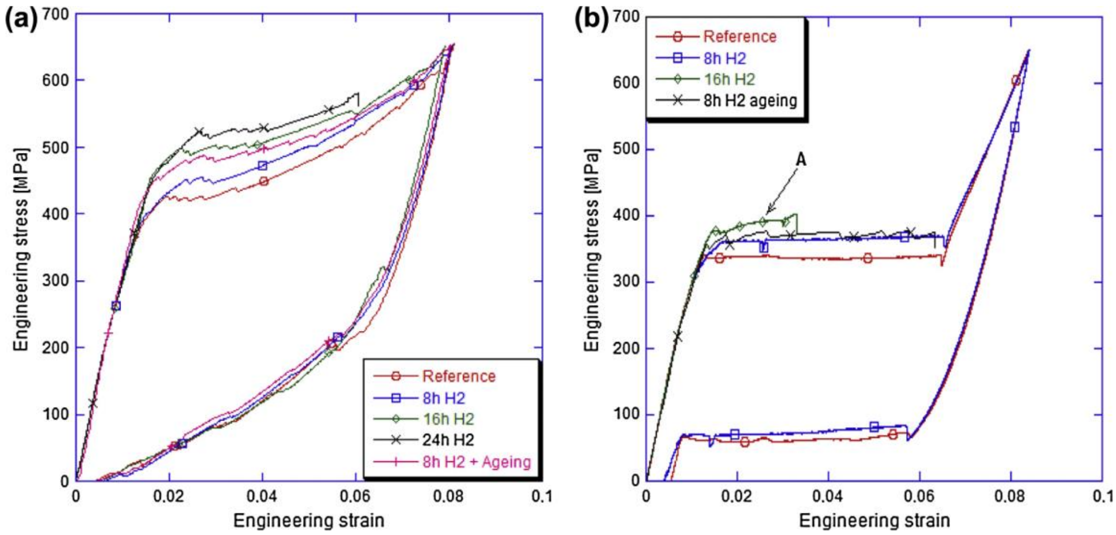

Figure 1: Stress–strain curves obtained immediately after 8–24 h of hydrogen charging and after 8 h of charging and ageing for 24 h at a strain rate of (a) 10 2/s and (b) 10 5/s [6]

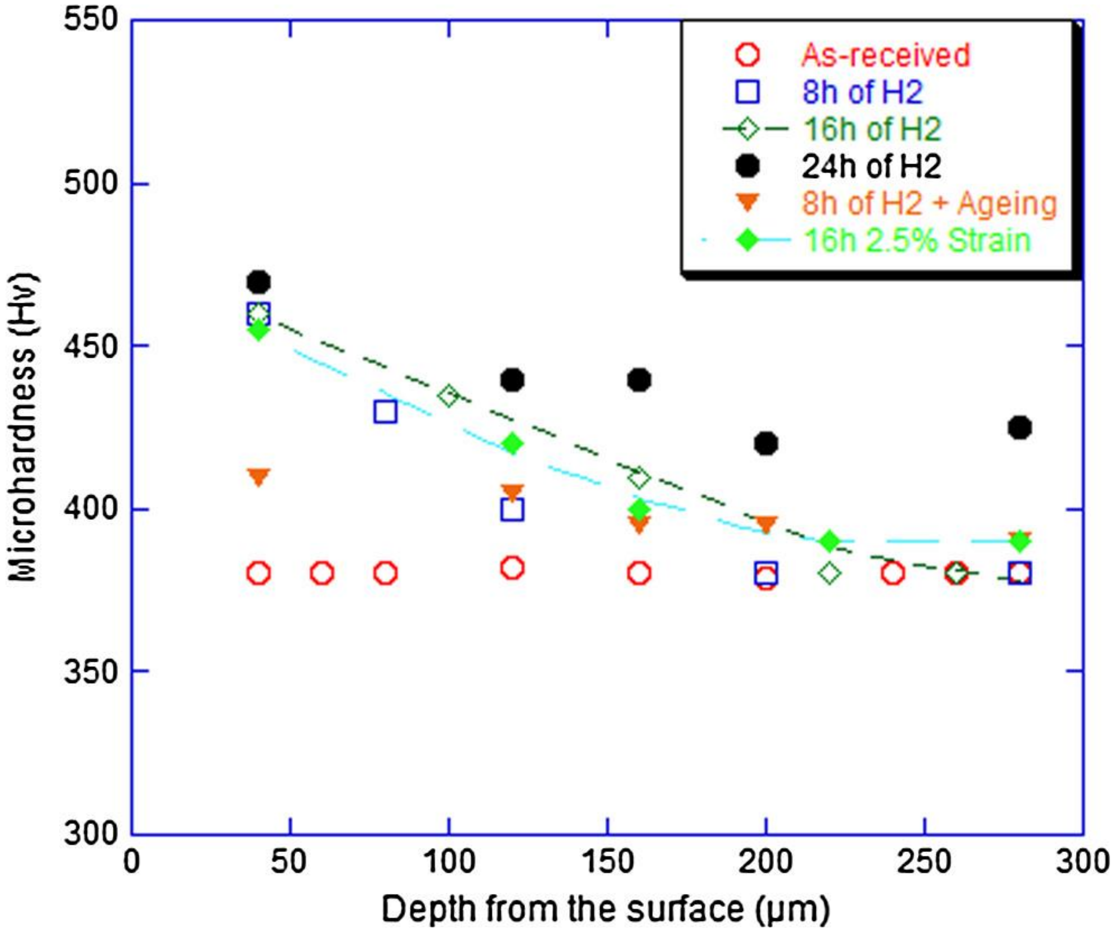

Figure 2: Vickers microhardness depth profile of the non-charged, as-immersed, and charged and aged specimens for 24 h specimens. Dashed lines show the Gaussian fit distributions of the as-charged, and the charged and strained until 2.5% specimens after 16 h [6]

2.6 Differential Scanning Calorimetry

Using a differential scanning calorimeter (DSC), the influence of ageing temperature on the reverse martensitic transition of the porous Ni-rich NiTi SMA is examined and discussed. Specimens were cut out of the aged materials in one section for DSC measurements. A differential scanning calorimeter (DSC) of type 2910 from TA Instruments was used to evaluate the reverse transformation behaviour during heating. The heating rates were kept constant at 5 degrees Celsius per minute. DSC specimens weighing between 20 and 50 mg were cooled to 60 °C and kept there for 3 minutes to achieve thermal equilibrium. The DSC measurement was then started by heating the sample to 60 °C. In Figs. 3 and 4, the DSC charts on heating of the samples aged at different temperatures for 1 or 2 hours are shown, respectively. Table 1 identified all temperatures that corresponded to the unique DSC peaks (peak temperatures) seen in

Figs. 3 and 4. In general, reverse martensitic transformations in dense Ni-rich NiTi SMAs on heating can be one-step (after solution treated) from B19’ to B2, two-step (after subsequent aged) from the formation of R-phase (B19’→R-phase, first DSC peak) followed by a transformation from R-phase to B2 (second DSC peak), or multiple-step (under certain ageing conditions) [21].

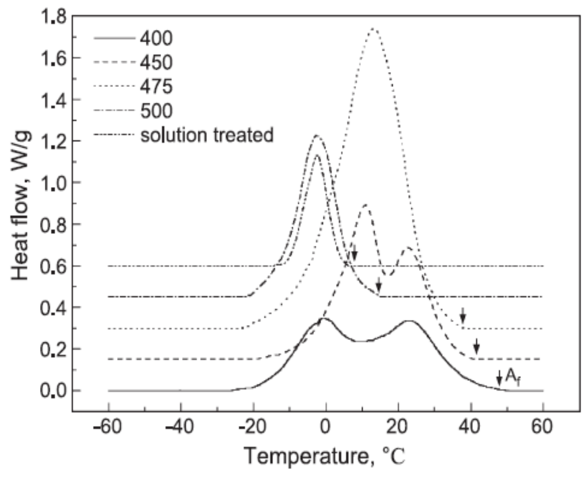

Figure 3: DSC charts on heating of porous Ni-rich NiTi SMA fabricated by SHS after solution treated at 1050 8C for 4 h and subsequently aged at different temperatures for 1 h. The small vertical arrows indicate the austenite finish temperature (Af) [21]

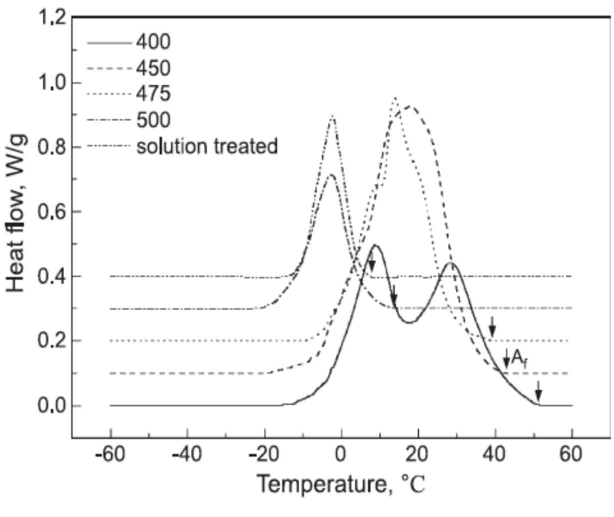

Figure 4: DSC charts on heating of porous Ni-rich NiTi SMA fabricated by SHS after solution treated at 1050 8C for 4 h and subsequently aged at different temperatures for 2 h. The small vertical arrows indicate the austenite finish temperature (Af) [21]

The reverse phase changes in porous NiTi SMA formed by SHS proceed significantly more slowly and even incompletely, as evidenced by the enlarged transformation span, modest slope, and weak intensity of the thermal peaks on the DSC curves in Figs. 3 and 4. As a result, the shape memory capabilities of porous NiTi SMA associated with the reversible martensitic transition have worsened [21].

2.7 NiTi Failure in Orthodontic Brackets

Orthodontic brackets are little metal or ceramic orthodontic attachments that are bonded to a tooth and used to anchor an archwire. Each attachment is either soldered or welded to a tooth-enclosing band or bonded directly to the tooth. Stainless steel, non-nickel or low-nickel stainless steel, and titanium brackets are the different types of metallic brackets. Since the nickel component in standard stainless steel has been shown to have genotoxic effects and may induce allergic reactions in patients, non-nickel or low-nickel stainless steels are used instead. This type of steel has a similar or better hardness than other steels, however it has a lesser corrosion resistance. Because of its improved biocompatibility and corrosion resistance, titanium has recently been employed as a bracket replacement [22].

Many orthodontic systems (for GDPs) go up to a 0.018in wire, and most adult orthodontic brackets have a bracket slot width of 0.022 inches. The wire is not only too short, but it's also the improper shape. So, in these circumstances, the bracket wire is straight, indicating that the wire has done its job, but the teeth are not aligned because the wire size is insufficient to cover the slot, indicating that there is some play in the system. If the wire used was thicker, say 0.021 inches in a 0.022 inch slot, and the wire was rectangular rather than round (say, 0.0210.025 Steel), these problems may be avoided.

The force-deflection curves of NiTi wire prior to bending at various deflections in the bracket system are shown in Figure 5. It can be observed that the curve's trend was influenced by the amount of deflection imparted to the wire during loading [23].

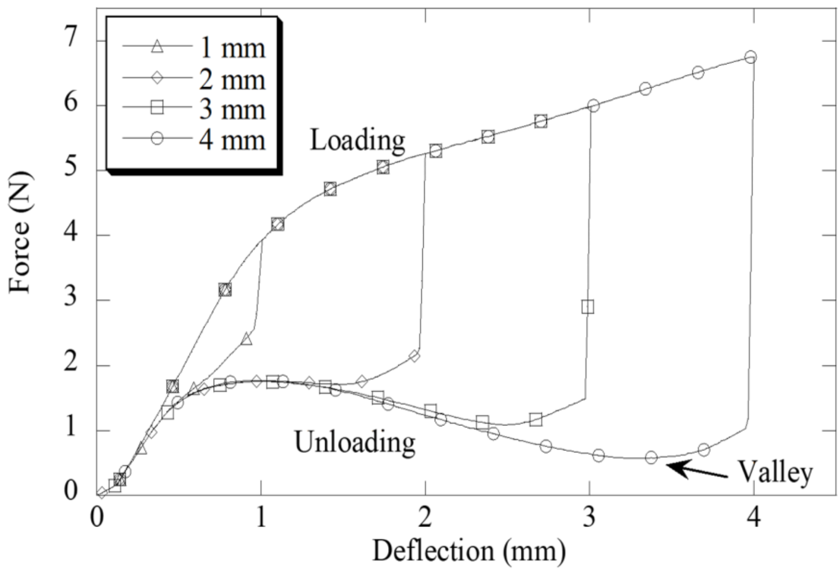

Figure 5: Force-deflection of NiTi wire at different magnitude of deflections [23]

When the NiTi wire was recovered from different magnitudes of deflections, it provided a distinct force-deflection curve, with lower unloading force being recorded for the wire loaded at larger deflection. The deformation of the wire began around the bracket corners during the bend, with the amount of primary stress and the volume of completely converted martensite steadily increasing in response to the applied deflection.

CHAPTER 3

METHODOLOGY

3.1 Introduction

As this project is moving into the initial stage, most effort was put in getting the orthodontic wire and planning the charging time and ageing time of the wire before going through the mechanical testing like Differential Scanning Calorimetry, bending test as well as tensile test. The type of wire used for the experiment is Nickel-Titanium rectangular natural super elastic wire (upper 0.016*0.022) as shown in Figure 1.

Figure 6: Nickel-Titanium rectangular natural super elastic wire (upper 0.016*0.022)

3.2 Experimental Setup for Hydrogen Charging and Ageing

Table 1: List of Apparatus

Apparatus	**Description**
Electrolyte	1% Sodium Sulphate (Na_2SO_4)
Anode	Stainless Steel plate (d = 25 mm)
Cathode	Orthodontic NiTi archwires
Power Supply	12V voltage, 224 μA
Multimeter	Set to DCA 2.5mA
Tripod stand	Hold the charging wires

The orthodontic NiTi archwires need to be cut into a straight wire. To prepare the electrolyte used in the hydrogen charging which 1 % of Na_2SO_4 aqueous solution, 3 g of sodium sulphate and 300 ml of distilled water needed to be mixed together. Before connecting the anode and cathode to the power supply, the stainless-steel plates need to be polish using 2000 grits sandpaper. The stainless-steel plates are connected to the positive terminal (anode) and the orthodontic NiTi archwire will be connected to the negative terminal (cathode).

After connecting the setup to the power supply, the connection needed to be check whether the current is flowing or not by observing the presence of bubbles produced at the cathode (NiTi archwire). The current is set to 224 μA using the ammeter or multi-meter. To get a more accurate results, digital ammeter can be used. The voltage used is 12 V and can be adjusted at the power supply board. The date and time of every hydrogen charging is recorded. For this project, the charging time will vary which are 8 hours, 16 hours and 24 hours.

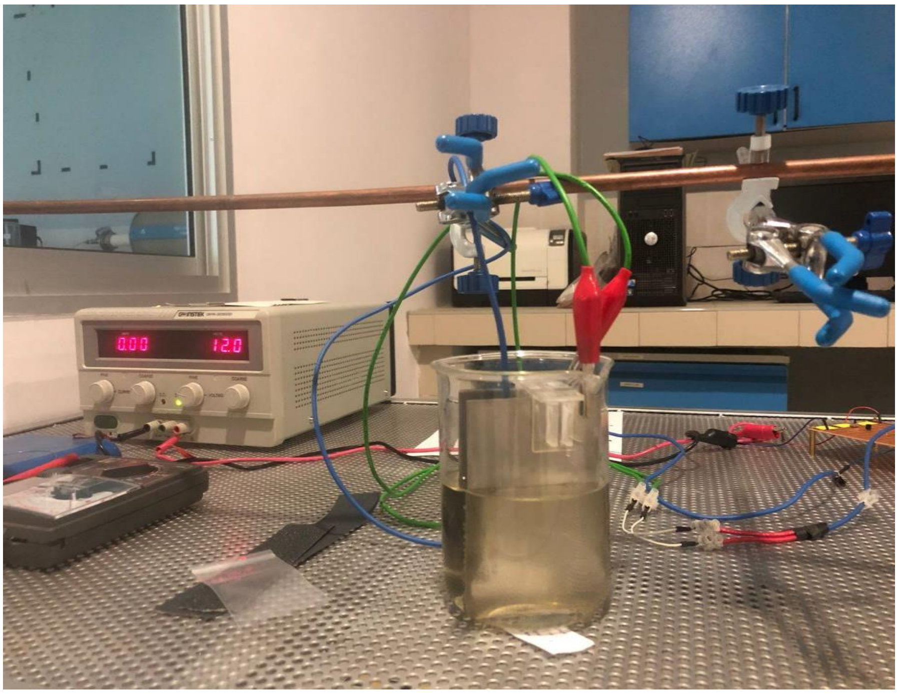

Figure 7: Experimental Setup for hydrogen charging

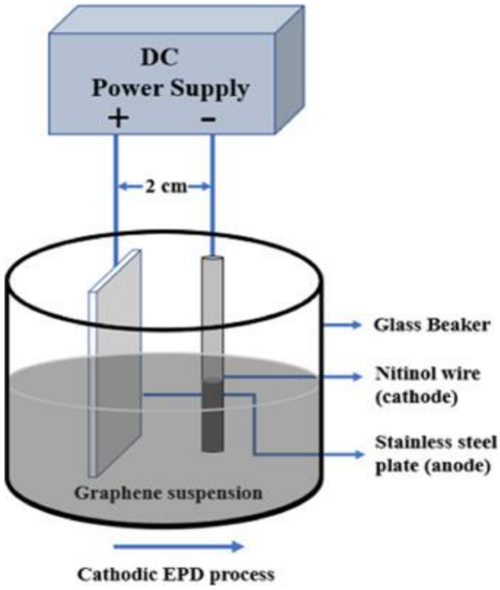

Figure 8: Schematic diagram of hydrogen charging into NiTi wire

Chemical equation for the hydrogen charging into the NiTi archwires:

$$2H_2O \rightarrow 2H_2 + O_2 \qquad (equation\ 1)$$

At the positive terminal (anode),

$$4OH^- \rightarrow O_2 + 2H_2O + e^- \qquad (equation\ 2)$$

At the negative terminal (cathode)

$$2H^+ + e^- \rightarrow H_2 \qquad (equation\ 3)$$

In order to study the interaction between ageing of the Nickel-Titanium wire after hydrogen charging, bending and tensile test have been carried out on as-charged specimens and aged samples at different time intervals in air at room temperature. The ageing period and charging period are as shown in the table below.

Table 2: Charging time and ageing time

Charging Time			
Ageing Time (day)	8 hours	16 hours	24 hours
1	/	/	/
2	/	/	
3	/	/	
4	/	/	/
5	/	/	/
6	/	/	
7	/	/	/
14			/

After the Nickel-Titanium wire has finished the hydrogen charging for 8 hours, 16 hours and 24 hours, the wire will be placed in a sealed bag to age for 1 to 7 days. The date, start time and end time for the process will be recorded on a piece of paper. As the wire finished the ageing process, it will be carried to the Applied Mechanics Laboratory and Nanofabrication and Functional Materials Laboratory to undergoes the Differential Scanning Calorimetry Analysis, 3-point bending, 3-bracket bending as well as tensile test using the Ultimate Tensile Machine.

3.3 Transformation Temperatures Determination using Differential Scanning Calorimeter (DSC)

The effect of hydrogen infusion in Nickel-Titanium orthodontic wire after ageing in air at room temperature is investigated using differential scanning calorimetry (DSC) and discussed. By using the DSC analysis, the transformation temperatures (M_f, M_s, R_f, R_s, A_s and A_f) can be measured. The heating and cooling rates are set to be 10 ˚C/min. The specimens were cut from the as-received wire into small pieces to fit in the crucible before measuring the weight of the specimen (5 mg -20 mg). After the specimen is placed in the DSC, the nitrogen gas flow needed to be on before using the TA instrument which is the software for using the DSC analysis.

In the TA instrument, the weight of the specimen is inserted and the method for the heat treatment needed to be set before using it. The method used in DSC for this wire is heat/cool/heat where the wire will be heated first until 100˚C. After that, the cooling chamber will be used to cool the specimen where liquid nitrogen is poured into the cooling chamber and cooled until -100˚C and will be heated again until 100 ˚C. Reset all run before running the software and the software will automatically stop when the heating process is done and changed into another specimen.

Figure 9: DSC analysis during cooling process using liquid nitrogen and cooling chamber

The program will automatically stop running when the heat treatment is done and the data for the phase transformation temperature is obtained. The data for the charged and aged wire will be compared to the as-received wire.

3.4 Three-Point Bending

When the wires had finished the charging and ageing process, it will immediately be tested at the Applied Mechanics Laboratory. The first test that was done is the three-point bending test. With the help of the technician, the correct load cell for the test was equipped. For these 3 different tests, different grips, load cells and jigs will be used. As for the 3-point bending test, the load cell, grip and jig are as shown in Figure 10.

Before starting the test, the sample will be placed on the jig and the grip need to be checked whether it touches the middle of the sample, if not it needed to be adjusted using the align key. Then, the UTM is connected to the computer to be able to use the software and run the tests. In the software, the specific method was chosen for the three-bracket test. The details of the specimen were inserted and cyclic test was done on the specimen. The raw data for the specimen was obtained.

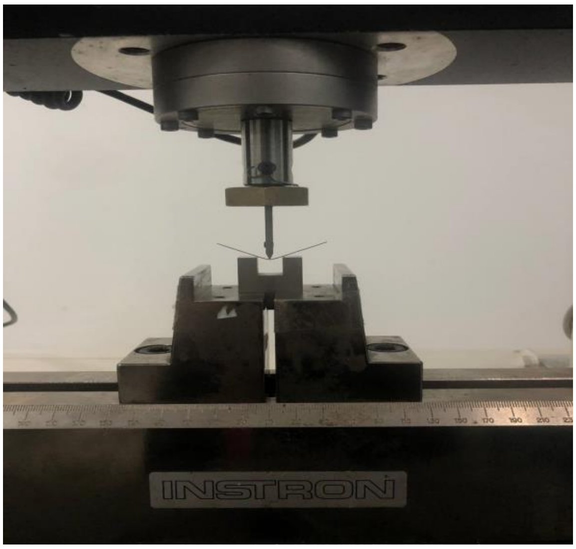

Figure 10 Jigs used for three-point bending (left) and three-point bending that was done on the orthodontic NiTi wire (right)

3.5 Three-Bracket Bending

After the Universal Testing Machine is connected to the computer, the universal testing machine's crosshead was fitted with the central bracket (which corresponded to the canine bracket). The lateral brackets (lateral incisor and first premolar) were attached to the three-bracket device' fixed portion. The interbracket spacing was fixed at 7.5 mm, measured from the middle of each installed bracket.

The brass mounts were positioned on a stainless-steel support and the three brackets were aligned utilising a complete bracket-slot stainless steel jig during the bonding technique (0.018 inch or 0.022 inch according to the vertical dimension of the tested bracket-slot). The jig was used to align the bracket-slot to guarantee that the bracket orientation was proper before executing each individual test after the brackets were joined to the three-bracket bending test equipment.

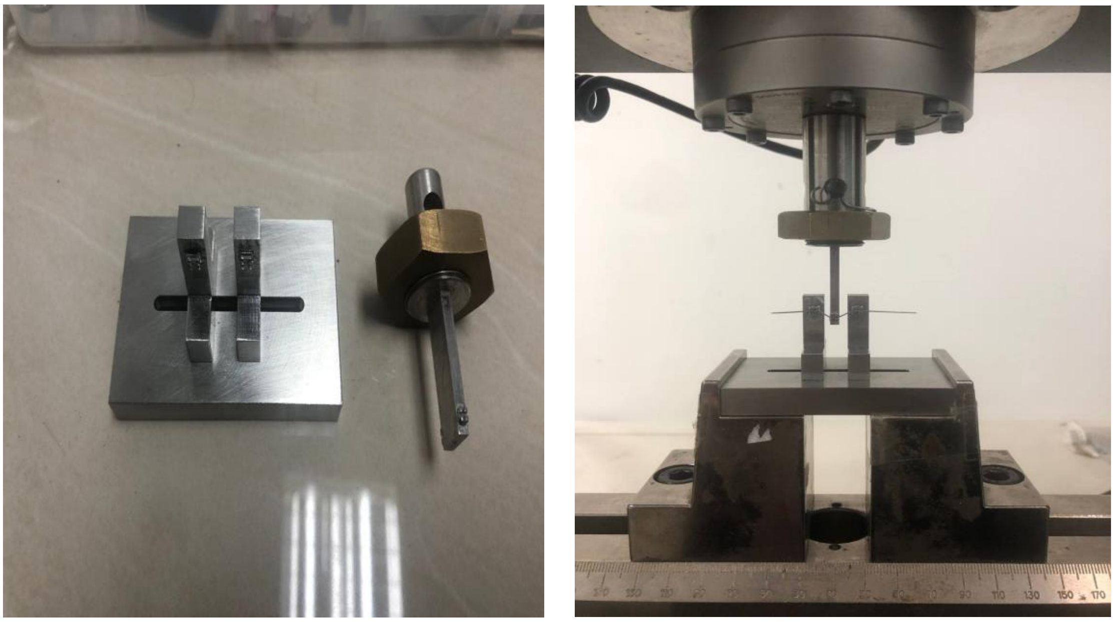

Figure 11: Bracket-slot stainless-steel jig (0.018 inch) (left) and bracket bending test on Nickel-Titanium Orthodontic wire (right)

In the software, the test method details can be customized such as the type of wire, the size of wire, the maximum extension. In this case, the type of wire is rectangular with the size of 0.4064 mm*0.5588 mm and the maximum extension for the test is set to 3.1 mm. The test is set to cyclic so that when the wire reaches maximum extension, the load that was applied to the wire will be reverted until it reaches 0 N.

3.6 Tensile Test

Different load cells and jig will be used in the tensile test to test the deformation behaviour of the NiTi orthodontic wire after it is charged and aged at the different times. The procedure of executing the tensile test was almost the same as doing the three-bracket and three-point bending test. The first thing that was done was setting up the method for the tensile test in the software. Before running the test, the load and extension value need to be reset. Cyclic test was done to obtain the results during loading and unloading of the NiTi wire.

Figure 12: Tensile test using the UTM

CHAPTER 4

RESULT AND DISCUSSION

4.1 Differential Scanning Calorimetry

Figure 13 shows the DSC curves of the as-received and aged NiTi wire specimens after hydrogen charging. The phase transformation temperatures of the specimen are marked with arrows for specimen aged for 14 days and 1 days. The austenite finish temperature (A_f) for the specimen aged for 14 days was approximately 19.1 ˚C. The specimen that was aged for 14 days and 1 days exhibited a single sharp peak during the cooling process and two sharp peaks during the heating process. As for the as-received specimen and specimen aged for 4 days, 5 days and 7 days, they exhibited a single peak which are not as sharp as the peaks from the specimen that were aged for 1 day and 14 days in both cooling and heating. In this case, hydrogen absorption on specimen that was aged for 14 days and 1 day show a better suppression effect especially during the M→A phase transformation during heating compared to A→M phase transformation during cooling.

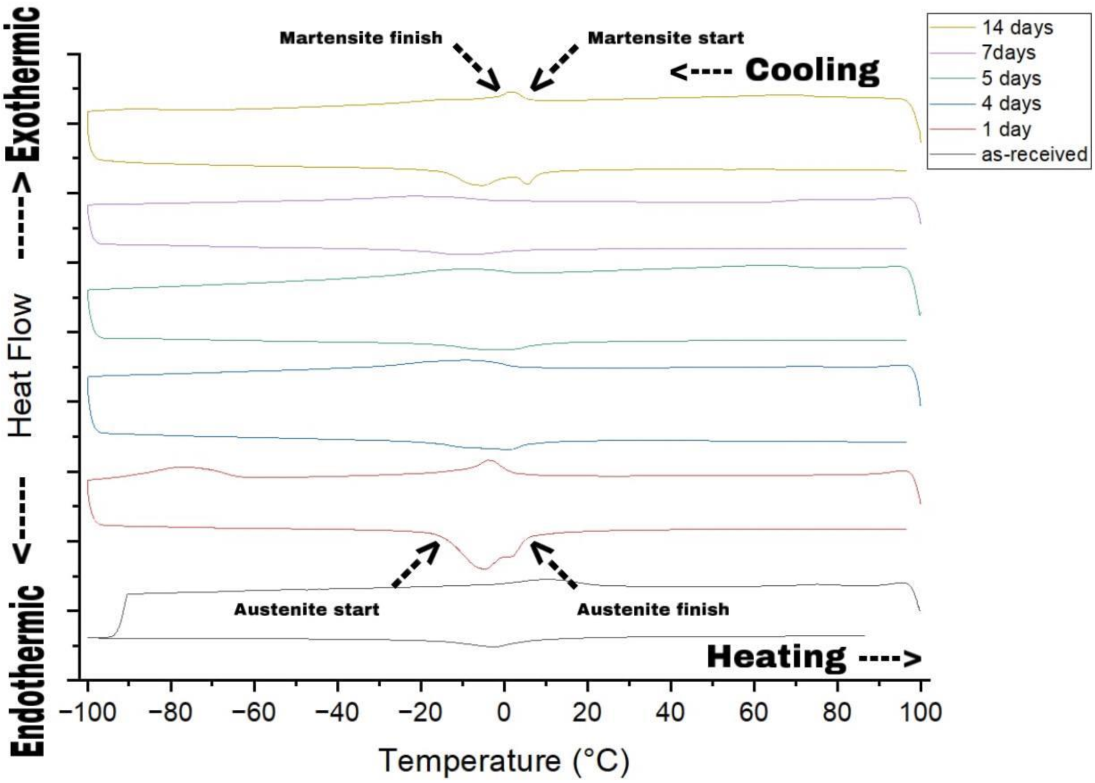

Figure 13: DSC Analysis curves for specimens charged with hydrogen for 24 hours

4.2 Three-Bracket Bending Test

Figure 14 shows the graph of bending force against deflection for the three-bracket bending test. The as-received specimen exhibited the highest value of bending force towards deflection of 3 mm compared to specimen charged with hydrogen and aged at room temperature. Specimen charged for 16 days and aged for 9 days could not recover fully after unloading leading to 1.33% residual strain from 10% deformation strain. As for specimen charged for 16 days and aged for 5 days, fracture occurred when the wire underwent deformation of 2.33 mm.

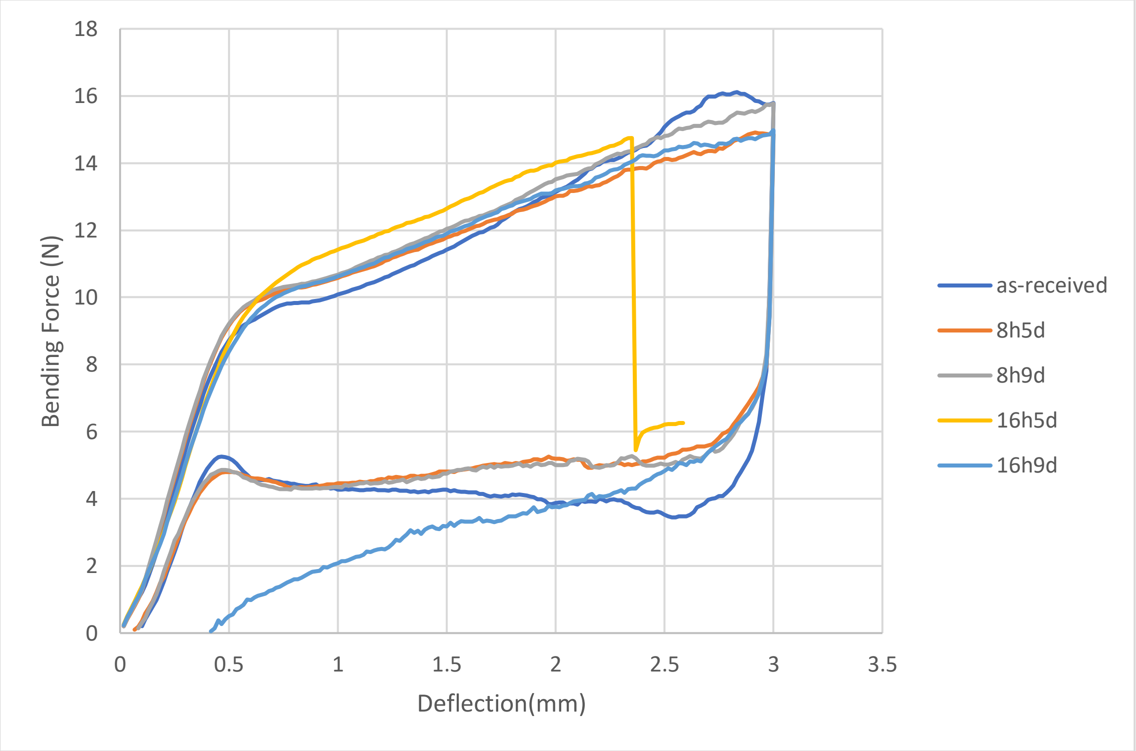

Figure 14: Three-bracket bending curves

Figure 15 shows the stress against strain for the three-bracket bending test for indicating the deformation behaviour of NiTi archwire specimen charged for 8 hours and 16 hours followed by aging at room temperature as well as the as-received specimen. It can be observed that specimen charged for 16 hours recorded the highest yield strength and the as-received recorded the lowest yield strength as compared to the other specimens. This shows that the longer the charging time, the higher the value of the yield strength obtained. The highest stress value was recorded by the as-received specimen and the lowest stress by specimen charged for 16 hours and aged for 9 days. As for specimen charged for 16 hours and aged for 5 days, fracture occurred at 7.8% strain before reaching 10% deformation strain.

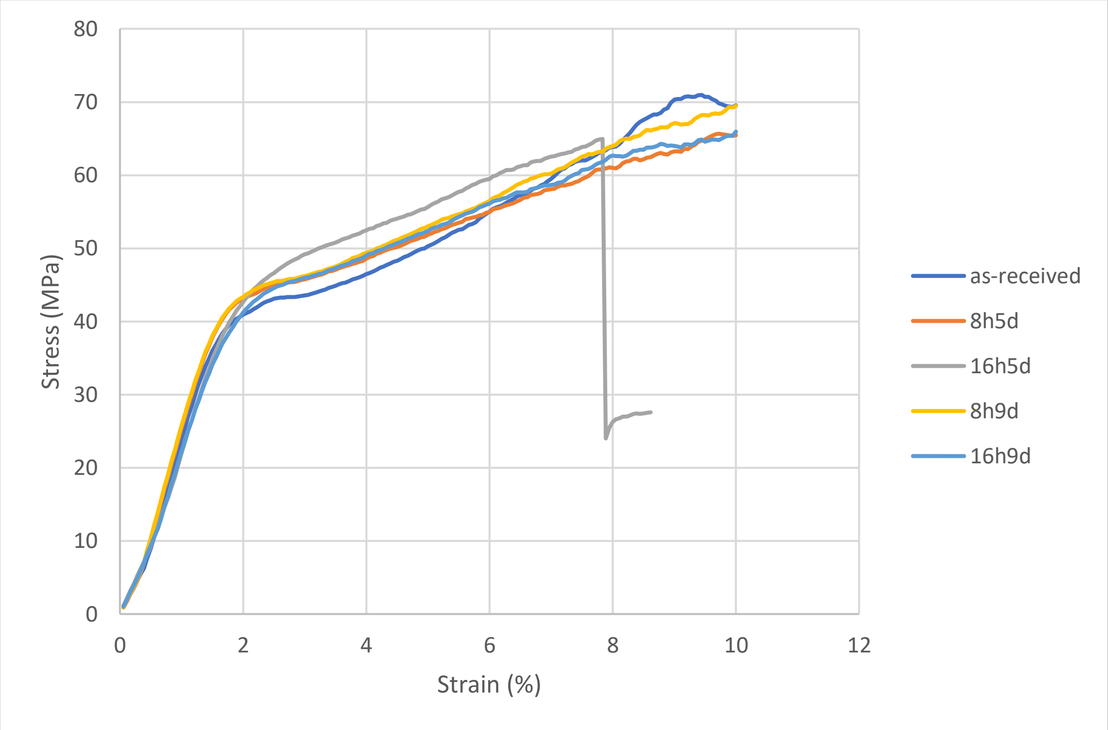

Figure 15: Graph of stress against strain for the three-bracket bending test

4.3 Three-Point Bending

Figure 16 shows the graph of bending force against deflection for the three-point bending test on the orthodontic NiTi archwires. The specimen used for this test are the as-received specimen, specimen charged for 8 hours and aged for 5 days and 9 days as well as specimen charged for 16 days and aged for 5 days and 9 days. After the deflection of 3.1 mm, for the as-received specimen and specimen charged for 8 hours, the wire returned to its initial length at 0 N. This indicates that it still conserved the ability to recover nearly fully after the unloading. Different case occurred for the specimen charged for 16 hours where it yielded 0.2 mm for the specimen aged for 5 days and 0.33 mm for specimen aged for 9 days.